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LSA LARGE AREA SILICON SHEET TASK
CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

QUARTERLY REPORT

APRIL - JUNE
1979

The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE.

siltec

Menlo Park, California

JULY 1979

LSA LARGE AREA SILICON SHEET TASK

CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

QUARTERLY REPORT #7
April - June 1979

George Fiegl

July 1979

SILTEC CORPORATION
Menlo Park, California

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ABSTRACT

This project, which is now in its second phase, is directed toward the design and development of equipment and processes to demonstrate continuous growth of crystals by the Czochralski method, suitable for producing single silicon crystals for use in solar cells. "Continuous" is defined as the growth of at least 150 kg. of mono silicon crystal, 150 mm. in diameter, from one growth container. The approach to meeting this goal is to develop a furnace with continuous liquid replenishment of the growth crucible, accomplished by a meltdown system with a continuous solid silicon feed mechanism and a liquid transfer system, with associated automatic feedback controls.

During Phase I of this program, it became apparent that silicon monoxide build up in the furnace interior was the major limiting factor to achieving mono crystalline growth for large portions of material processed. As a result, growth under reduced pressure is a major goal of the program plan under Phase II. Furnace conversion for operation in low pressure range was completed during the last quarter.

Batch recharging of the meltdown chamber, as exercised under Phase I of this program, prohibited taking full advantage of a continuous process. Our current effort included development of systems for continuous solid recharging of the meltdown chamber for various forms of poly silicon. Design concepts and fabrication of one system are complete.

The melt transfer system used during Phase I is further developed to increase its lifetime significantly, and at the same time, drastically improve the economics of the system. Latest efforts include the design of a new heating element and a reduction of the total melt transfer length.

To acheive the economic goals for 1986, the Continuous Liquid Feed (CLF) Furnace must be capable of producing crystals of 150 mm. in diameter, with solidification rates $>$ 2 kg./hr. and a minimum run throughput of 150 kg.

CONTENTS

1.0 INTRODUCTION

The overall purpose of this program, which began in October 1977, is to demonstrate continuous crystal growth by the Czochralski method. Continuous is defined as a throughput of silicon that produces 150 Kg of single crystal material, 150 mm in diameter, from one common container.

The program of Phase II includes:

- Furnace conversion for low pressure operating conditions.
- Design, development and fabrication of continuous solid feed systems for silicon of various forms, for the meltdown chamber.
- Design, development and fabrication of the continuous liquid silicon feed system with a reuseable heater and automatic feedback controls.
- Demonstration of continuous crystal growth as defined above, with an after growth yield $>$ 80%.
- Definition of process control variables for continuous Czochralski growth.
- Theoretical analysis of silicon sheet growth to be considered during design and development of the furnace.

The design changes and the conversion of the furnace to low pressure operation was accomplished during the last quarter. Design fabrication of a poly rod feed mechanism to the meltdown chamber and the conceptional designs for continuous feed mechanisms were completed. The transfer tube heating element development for reuse represents a major part of this quarter's efforts.

2.0 RESULTS

2.1 GROWTH UNDER LOW PRESSURE

During Phase I of this program, several 100 Kg throughput runs were attempted, with the best effort achieving 70 Kg of grown ingot. A major limiting factor was the silicon monoxide buildup in the furnace interior over longer periods of time (50 to 60 hrs). Particular problem areas were around the transfer tube from which particles of monoxide transfered into the melt and disturbed monocrystalline crystal growth. To reduce silicon monoxide buildup in critical areas and minimize argon consumption growth under low pressure became a major part under Phase II of this program. All dynamic and static seals were converted to vacuum seals so that a total systems leak rate of 10^{-4} torr liters/sec was achieved. A valving system for independent evacuation of the S.S. bellows independent from the main chamber was introduced to allow crystal removal while the furnace is under operating condition. The design for a coaxial purge system (See Page 9, Figure 1) for low pressure operation was completed. Argon is directed through a purge tube towards the melt surface and leaves the furnace through a tube centered around the purge tube. Silicon-monoxide emenating from the melt surface is immediately carried out through the argon purge gas and transported into the exhaust system, before a major distribution in the furnace interior can occur. Dimensions and locations of the concentric tubes are very critical to achieving optimum purge results.

The operating pressure toward the low end is limited through the vapor pressure of molten silicon and the transfer tube design. Both chambers are operated under the same pressure with melt transfer achieved through a small hydrostatic pressure difference.

2.2 POLY ROD FEED MECHANISM

To determine system impurity build-up in the growth crucible, the simplest form of solid feed mechanisms was developed. (See Page 10, Figure 2)

Poly rods 6" in diameter up to 50 Kg (presently the largest single poly rods available) are lowered into the meltdown crucible through a feedback control system, with the melt level sensor of the growth crucible as input, whereby melting and solidification in the growth chamber are kept in balance.

After a poly rod is completely melted and a crystal of equal weight grown, a new poly rod can be introduced while the crystal in the bellows is cooling down. This procedure can be repeated several times, and is only limited by the dissolution rate of the quartz liners. The major advantage of continuous solid feeding is the small melt volumes in both meltdown and the growth crucible, and relatively small material losses in case of furnace failure.

The design and fabrication of parts was completed during this period.

2.3 CONTINUOUS "LUMP" FEED MECHANISM

A parallel effort to design a solid feed mechanism for silicon lumps to the meltdown crucible is being made.

Due to the nature of the melt transport mechanism to the growth crucible, the weight of the individual silicon

pieces that can be introduced into the meltdown crucible at any one time is limited to approximately 50 grms to avoid severe melt level changes in the growth chamber and to simplify control circuits.

Silicon particles are transported through vibratory feeders into the meltdown chamber, as silicon solidifies at the same rate in the growth chamber.

One of the major design criteria for this system is to avoid contamination of the poly silicon through container materials. The program plan calls for design and fabrication of the particle feed system. Testing under operating conditions is planned for the first quarter of 1980.

2.4 REUSEABLE TRANSFER TUBE HEATER

Problems with heater life time over a period of 60 to 70 hrs during earlier test runs prompted a redesign effort of the heating element. (See Page 11, Figure 3) Two heating elements surrounding a 25 mm quartz tube of U shape are connected in series. The element is made out of graphite and supported by a ceramic insulator that also provides thermal insulation. The entire system is then encapsulated by silicon carbide shells. Molybdenum metal strips are used to form electrodes.

This system allows reuse of all parts with the exception of the quartz tube. The required power input for the 30" long heated quartz tube is 1.5 KW.

3.0 CONCLUSIONS

3.1 Operating the CLF furnace under low pressure with an appropriate exhaust system reduces silicon monoxide in the furnace interior significantly, and improves the economics of the system through a reduction in argon usage.

3.2 Continuous solid feed mechanisms for the meltdown chamber will significantly improve the productivity of the CLF furnace, and make the furnace operation simpler. Repriming of the transfer tube, which was required for batch recharging is eliminated. The temperature of the meltdown crucible can be kept constant throughout the run, and thereby minimize the devitrification of the quartz crucible.

3.3 Transfer tube system is limiting the operating furnace pressure towards the low side due to the distance between the melt level in the crucibles and the horizontal section of the transfer tube.

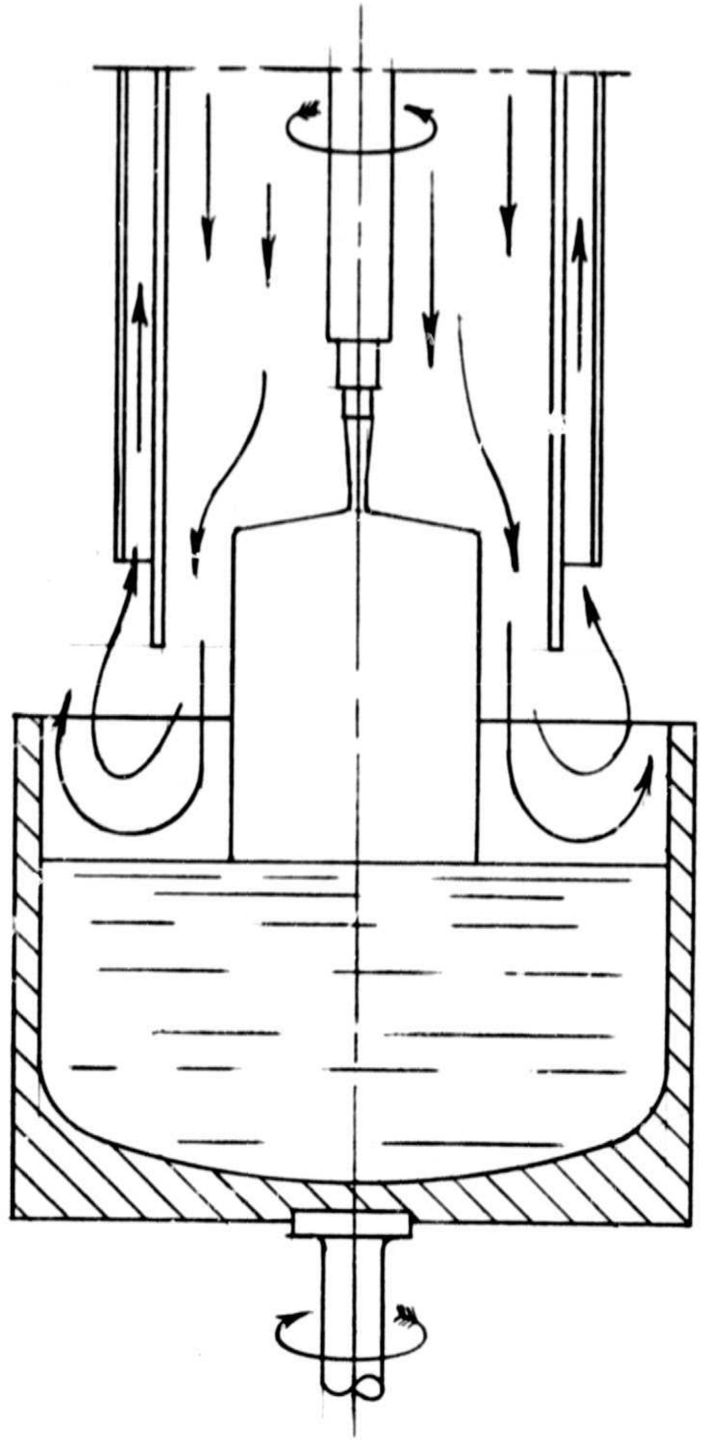
4.0 PROJECTION OF ACTIVITIES

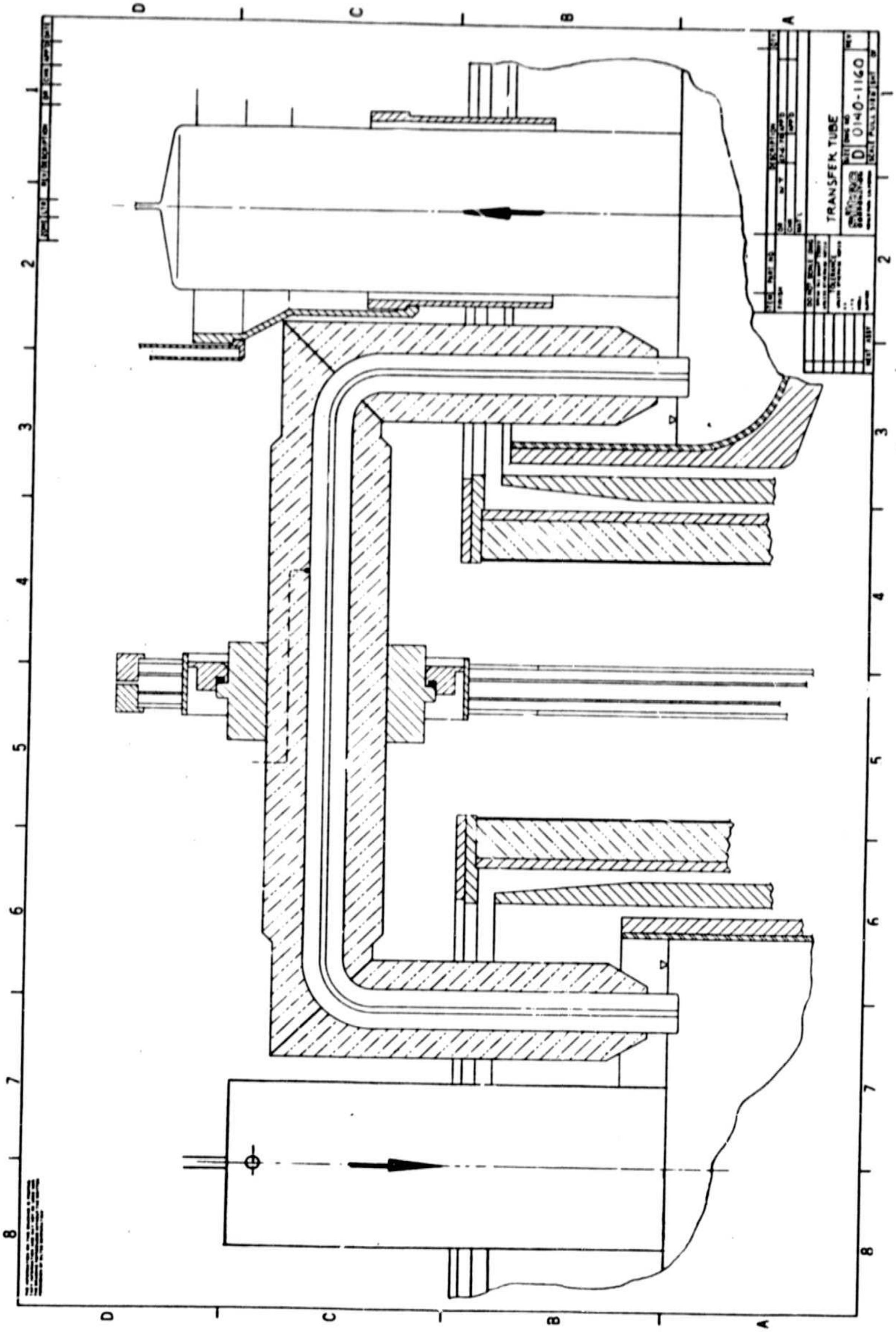
Several single runs by conventional CZ method are planned to establish basic furnace performance for 6" diameter ingots.

All other activities are following the program plan.

APPENDIX I

COAXIAL PURGE SYSTEM

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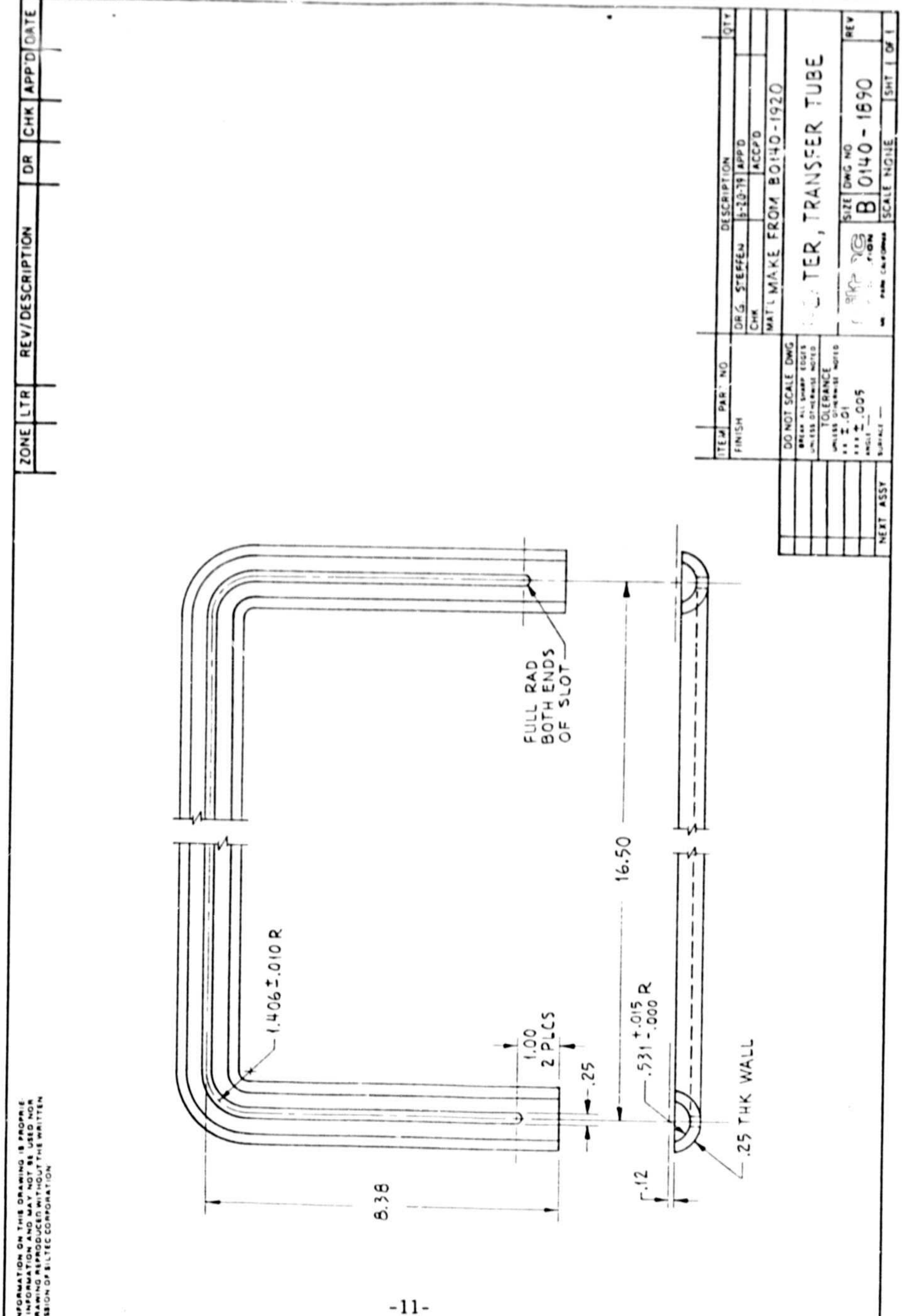
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1.00 2 PLCS
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.25 THK WALL
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8.38
THE INFORMATION ON THIS DRAWING IS PROPRIETARY INFORMATION AND MAY NOT BE USED NOR THE DRAWING REPRODUCED WITHOUT THE WRITTEN PERMISSION OF SILTEC CORPORATION
ITEM PART NO DESCRIPTION QTY
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DRG. STEFFEN 6-20-79 APP'D
CHK ACCP'D
MAT'L MAKE FROM B0140-1920
DO NOT SCALE DWG
BREAK ALL SHARP EDGES UNLESS OTHERWISE NOTED
TOLERANCE UNLESS OTHERWISE NOTED
.xx ± .01
.xxx ± .005
ANGLE —
SURFACE —
TER, TRANSFER TUBE
SIZE B DWG NO 0140-1890 REV
SCALE NONE SHT 1 OF 1
NEXT ASSY

APPENDIX II

NEW TECHNOLOGY

No new technology was generated during the reporting period.

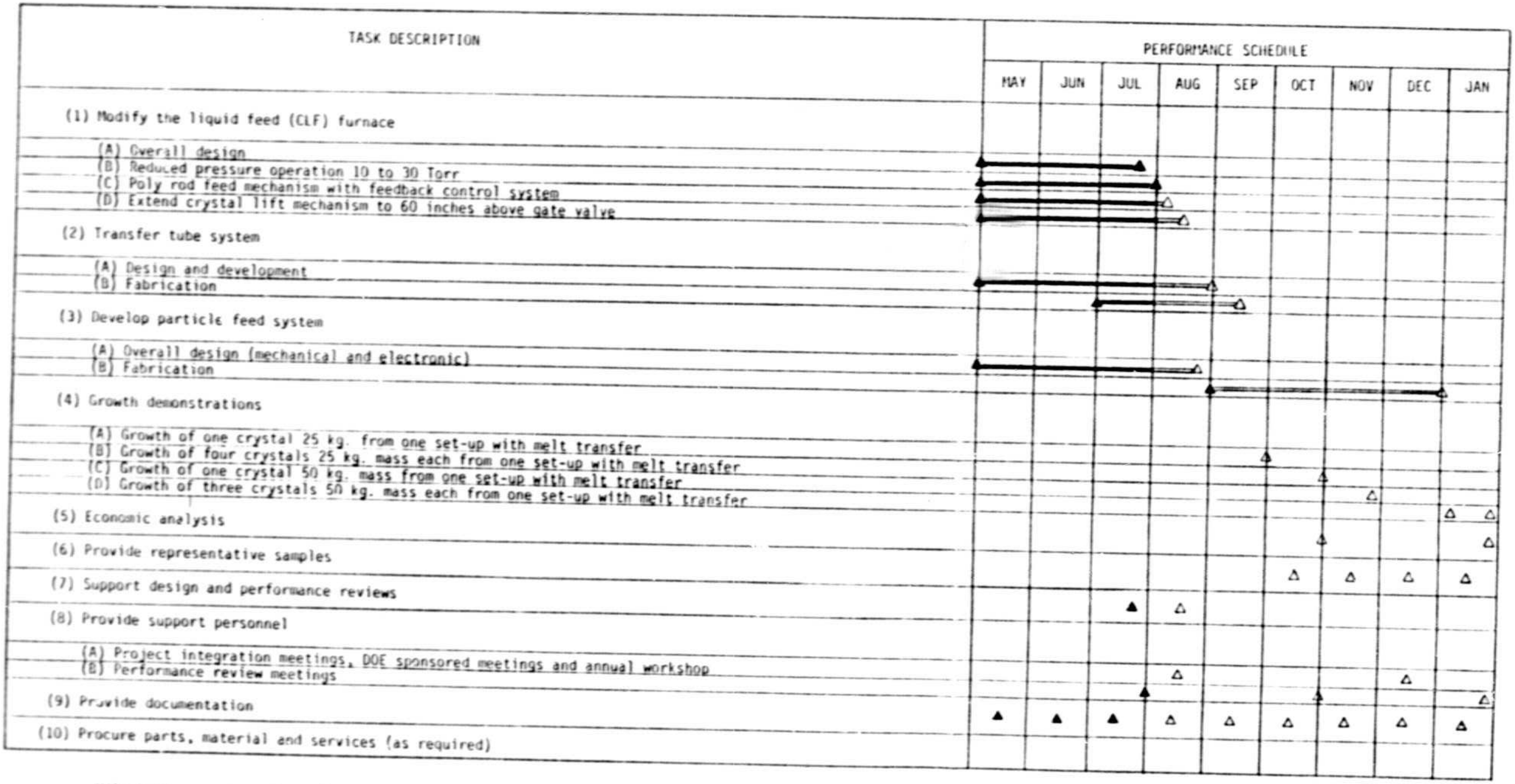
APPENDIX III

Summary of Characterization, Design, and Economic Analysis Data

Now new data were generated during this quarter to analyze.

APPENDIX IV

Milestones
Labor Summary
Cost Summary

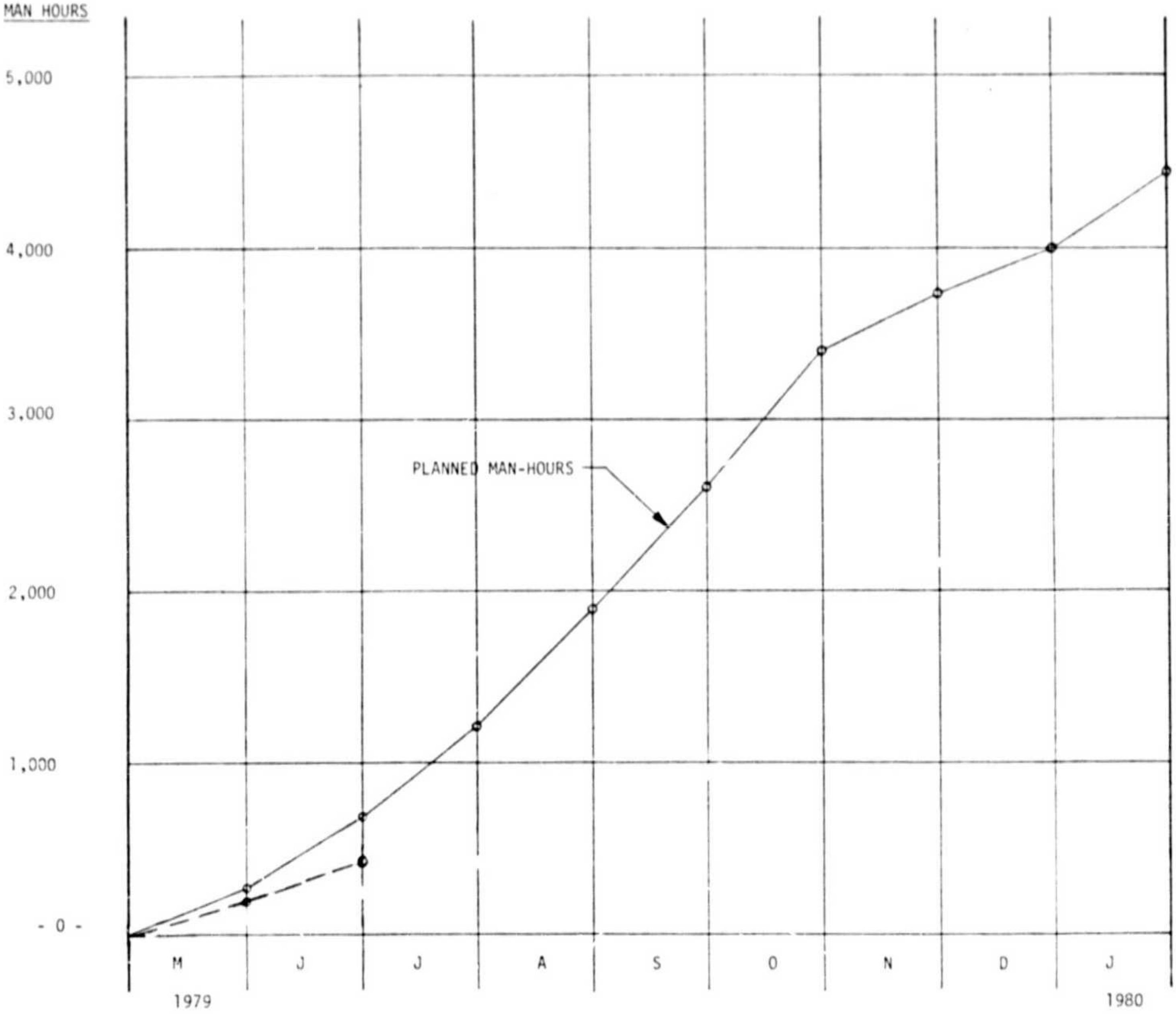


Milestone Legend △ Due Date △——△ Start-Finish

LSA LARGE AREA SILICON SHEET TASK

CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

PLAN VERSUS ACTUAL MAN HOURS



	Man-Hours
Previous Total	200
June 1979 Total	211
Total to Date	411

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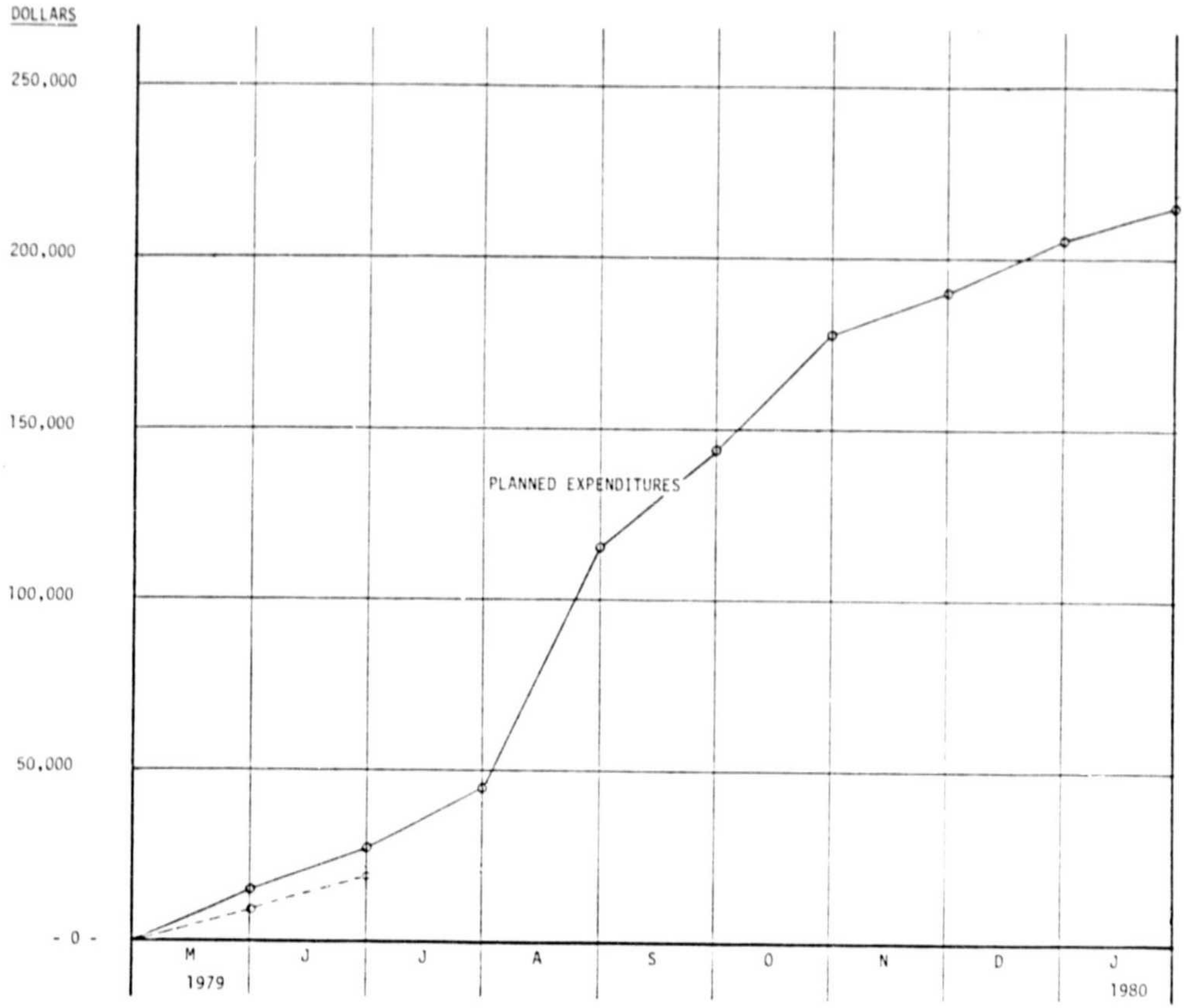
CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

PLAN VERSUS ACTUAL EXPENDITURES



	Projected Costs
Previous Total	$ 7,193.92
June 1979 Total	12,073.44
Total to Date	19,267.36